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DEVELOPMENT OF AN ELECTROPHORETIC IMAGE DISPLAY

QUARTERLY TECHNICAL REPORT

August 1 to October 31, 1981

Sponsored by

DEFENSE ADVANCED RESEARCH PROJECTS AGENCY

DARPA Order No. 3802

Contract No. MDA903-79-C-0439

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Contract Period: 20 Sept. 1979 - 30 Sept. 1981

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Prepared by

PHILIPS LABORATORIES

A Division of North American Philips Corporation
Briarcliff Manor, New York 10510

December 1981

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM	
1. REPORT NUMBER	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER	
	AD-A10924		
4. TITLE (and Subtitle)		5. TYPE OF REPORT & PERIOD COVERED	
DEVELOPMENT OF AN ELECTROPHORETIC IMAGE DISPLAY		Quarterly Technical Report August 1 to October 31, 1981	
		6. PERFORMING ORG. REPORT NUMBER	
		8. CONTRACT OR GRANT NUMBER(s)	
7. AUTHOR(s) Richard Liebert Joseph Lalak Richard Stolzenberger		MDA903-79-C-0439	
9. PERFORMING ORGANIZATION NAME AND ADDRESS PHILIPS LABORATORIES A Division of North American Philips Corp. Briarcliff Manor, New York 10510		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS DARPA Order No. 3802	
11. CONTROLLING OFFICE NAME AND ADDRESS Defense Advanced Research Projects Agency 1400 Wilson Boulevard Arling, Virginia 22209		12. REPORT DATE December 1981	
		13. NUMBER OF PAGES 19	
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report) UNCLASSIFIED	
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE	
16. DISTRIBUTION STATEMENT (of this Report) APPROVED FOR PUBLIC RELEASE DISTRIBUTION UNLIMITED			
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)			
18. SUPPLEMENTARY NOTES			
19. KEY WORDS (Continue on reverse side if necessary and identify by block number)			
electrophoretic image display		indium oxide	
control-grid electrode		matrix-addressed display	
ion-beam milling		sealing	
		display drive circuits	
20. ABSTRACT (Continue on reverse side if necessary and identify by block number)			
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PREFACE

This work is being performed by Philips Laboratories, a Division of North American Philips Corporation, Briarcliff Manor, New York under the overall supervision of Dr. Barry Singer, Director, Component and Device Research Group. Mr. Richard Liebert, Metallurgist, is the Program Leader; Mr. Joseph Lalak, Electronic Engineer, is responsible for cell fabrication and technology. Mr. Karl Wittig, Electrical Engineer, is responsible for circuit design; Dr. Howard Sorkin, Organic Chemist, is responsible for electrophoretic suspensions; Mr. Richard Stolzenberger, Physicist, is responsible for the Phase II technology.

This program is sponsored by the Defense Advanced Research Projects Agency (DARPA) and was initiated under Contract No. MDA903-79-C-0439. Dr. Robert E. Kahn is the Contracting Officer's Technical Representative for DARPA.

The work described in this ninth Quarterly Technical Report covers the period from 1 August to 31 October 1981.

SUMMARY

The purpose of this work is to develop an X-Y addressed electrophoretic image display. Redeposit of indium oxide onto the walls of the potential wells formed by ion-beam milling has delayed delivery of Phase I displays suitable for evaluation by ISI. Methods for removing this redeposit were only partially successful, and means of preventing it are being sought. Several Phase I displays were successfully demonstrated within Philips; one was fabricated using the photopolymer technology being developed for Phase II. This and other devices using the photopolymer dielectric showed much less sticking of pigment and therefore improved contrast, than that of the ion-beam-milled Mylar devices. Other photopolymers are being evaluated and perimeter sealing methods compatible with photopolymer construction are under investigation.

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1. INTRODUCTION

Several of the Phase I displays fabricated during this quarter were demonstrated within Philips. The devices were primarily of the ion-milled Mylar type but one was made using the photopolymer technology being developed for Phase II.

Ion-beam milling has resulted in the redeposit of In_2O_3 onto the Mylar walls of the potential wells. This redeposit can result in unsatisfactory performance due to the reduced resistance between the row and column electrodes. Some success in removing this redeposit has been achieved, and techniques for preventing redeposition are being investigated.

With the photopolymer technology being developed for Phase II, redeposit cannot occur. Devices fabricated in this way showed greater contrast and did not have the reduced row-to-column electrode resistance noted in the ion-milled devices.

A number of photopolymers and methods of coating them uniformly to the desired thickness are under consideration. Suitable materials and deposition methods for the control electrodes are being evaluated. An alternate technique for making the perimeter seal is being investigated.

2. PHASE I

2.1 Fabrication Technology

High-quality, row-electrode substrates continue to be fabricated by ion-beam milling. Commercial tin-doped In_2O_3 coated glass to which additional In_2O_3 has been added by reactive evaporation continues to be used.

Reactive evaporation of In_2O_3 is also being used for depositing the control electrode onto the Mylar dielectric. As discussed in Section 2.3, it has been necessary to use wet chemical etching of completed control-electrode structures.

To improve the resistance of the In_2O_3 to these etchants, we are investigating the role of substrate temperature and O_2 partial pressure during deposition on the film properties.

Increased substrate temperature has resulted in films which are more transparent without loss of conductivity and are compatible with the etchants. These films also have a reduced number of pinholes as a result of the way the crucible is now loaded and improvement in the predeposition heating cycle. Investigation of the In_2O_3 deposition system will continue especially with regard to establishment of optimum deposition conditions for use with the Phase II photopolymer technology.

Ion-beam milling continues to be used to form the wells in the Mylar dielectric. However, the redeposit of In_2O_3 onto the walls of the wells will make it necessary to modify the present process. These modifications are described in Section 2.3.

2.2 Device testing

An automated test facility has been set up to measure the resistance between the various electrodes on devices in process or on completed displays. To remain within the 40 terminal limitation of the test set, the row electrodes are connected in parallel in groups of approximately 10 for a total of 22 row groups. The column electrodes are similarly connected into 14 groups. The four redundant anode connections bring the total to 40. The software controls the series of tests, and the results are tabulated and printed out. The present tests include: verification of anode contact, resistance measurement between each row or column group and the anode, resistance between adjacent row groups, resistance between adjacent column groups, and resistance between each row group and each column group. This system has been used extensively to evaluate cells fabricated during this quarter. Substrates free of row-electrode shorts are being fabricated routinely; shorts to the anode are

uncommon. The most significant finding is that the resistance between a typical row group and a typical column group is often between $10^5 \Omega$ and several times $10^6 \Omega$ rather than greater than $100 \times 10^6 \Omega$ as expected.

Previous testing with multimeters failed to show this low row-to-column resistance. Such meters apply only about 1.5 V to the unknown in resistance measurements. The automated test set provides a selectable test voltage. Only when measurements were made at voltages typical of those used in operation of a completed device was this problem evident. Below about 20 V the row-to-column resistance appeared high but it dropped to much lower values at higher voltages.

These results could be obtained on either cells which had not yet been filled or on those which contained a suspension. Thus, the presence of conductive contaminants in the suspension has been ruled out.

This low row-to-column resistance hampered evaluation of completed devices under operating conditions, since the voltage between rows and columns is typically 15 V to 50 V. The problem was particularly troublesome with the portable driver. This driver is constructed in such a way that corresponding pixels in each of the 512 characters are connected in parallel. This results in even lower row-to-column resistance and loading of the power supplies in the driver. When the better devices were tested in a simpler driver with fewer pixels in parallel, good results were obtained. Simple test patterns having good contrast were achieved with the expected writing voltages.

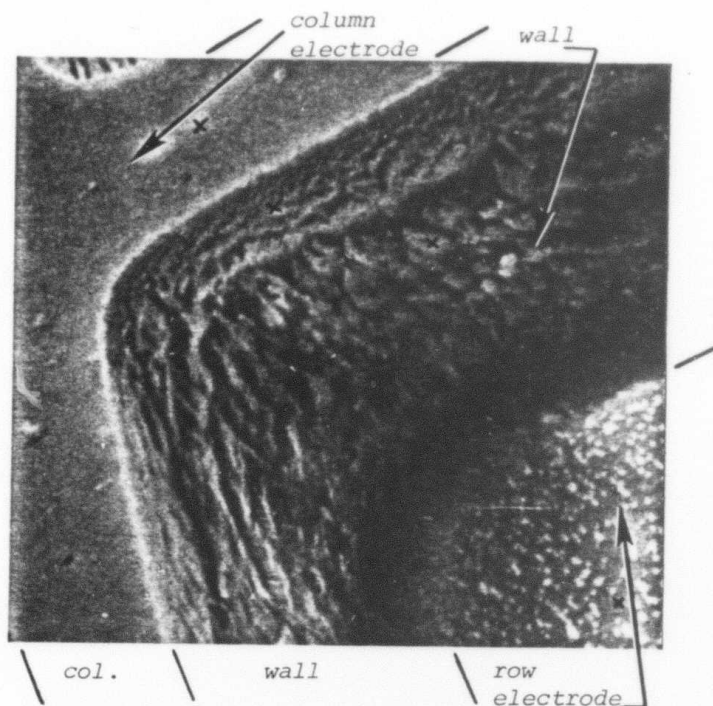
Several of the devices fabricated recently were successfully operated on the portable driver. Writing times less than 10 ms could be obtained with addressing voltages less than 50 V. The suspensions described in the previous Quarterly Report were used and sticking was not a significant problem. These devices were

used in a demonstration of EPID technology to executives and managers within Philips.

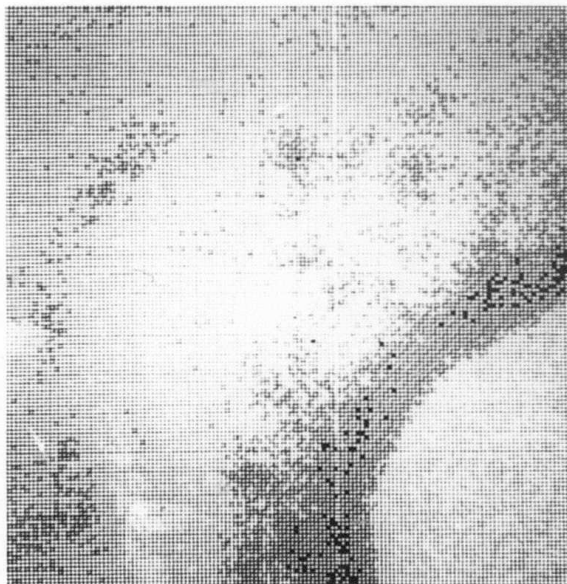
2.3 Redeposit of In_2O_3

The low row-to-column resistance noted in Section 2.2 is now known to be due to the redeposit of In_2O_3 onto the walls of the wells during ion-beam milling. Figure 1 shows some of the evidence for this conclusion. Scanning Auger Microscopy (SAM) was used to analyze the surfaces of various portions of a number of samples. SAM can identify and quantify the elements present in a 10 Å to 30 Å thick layer at the surface of a sample. Figure 1a is a secondary electron detection image of a corner of a well in a device in which the high voltage resistance between typical row and column groups was several megohms. The row electrode is tin-doped In_2O_3 which was not overcoated with evaporated In_2O_3 . Note the appearance and texture of the wall. SAM maps of the same area appear in Figures 1b and 1c. In this case, the brightness of the image is proportional to the amount of a particular element present. In this case, Figures 1b and 1c are for indium and carbon, respectively. Note that there is a discontinuous distribution of indium along the wall. As expected, indium is also detected on both the row and column electrodes. Where the indium on the walls is very thin or absent, the carbon signal from the Mylar is strong. The absence of carbon on part of the wall indicates an indium deposit thicker than the 10 Å to 30 Å analysis depth. The presence of carbon on the column electrode is probably caused by the lift-off photoresist.

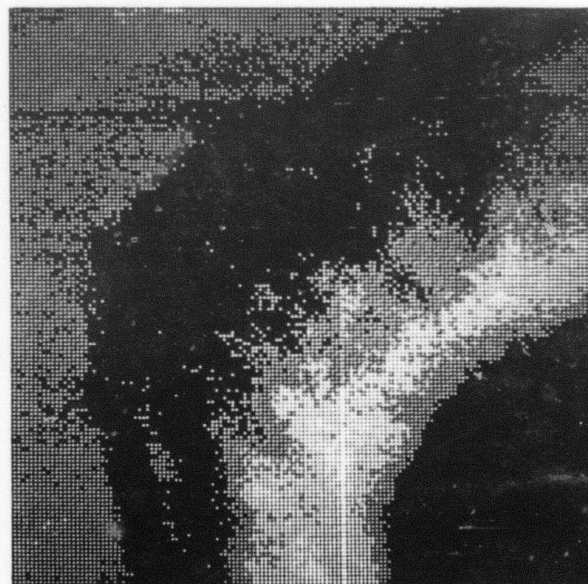
Figure 2 shows the Auger Electron Spectrum (AES) for a point on the wall of the sample. From the AES we see that only carbon, oxygen, indium, tin, and chlorine are present on the surface. The chlorine remains from cleaning the sample in trichloroethylene prior to analysis.



(a) Secondary electron detection image showing the column electrode, wall, and row electrode at the corner of a well.



(b) Map of indium distribution in same area as (a).



(c) Map of carbon distribution in same area as (a).

Figure 1: Photographs from scanning Auger analysis.

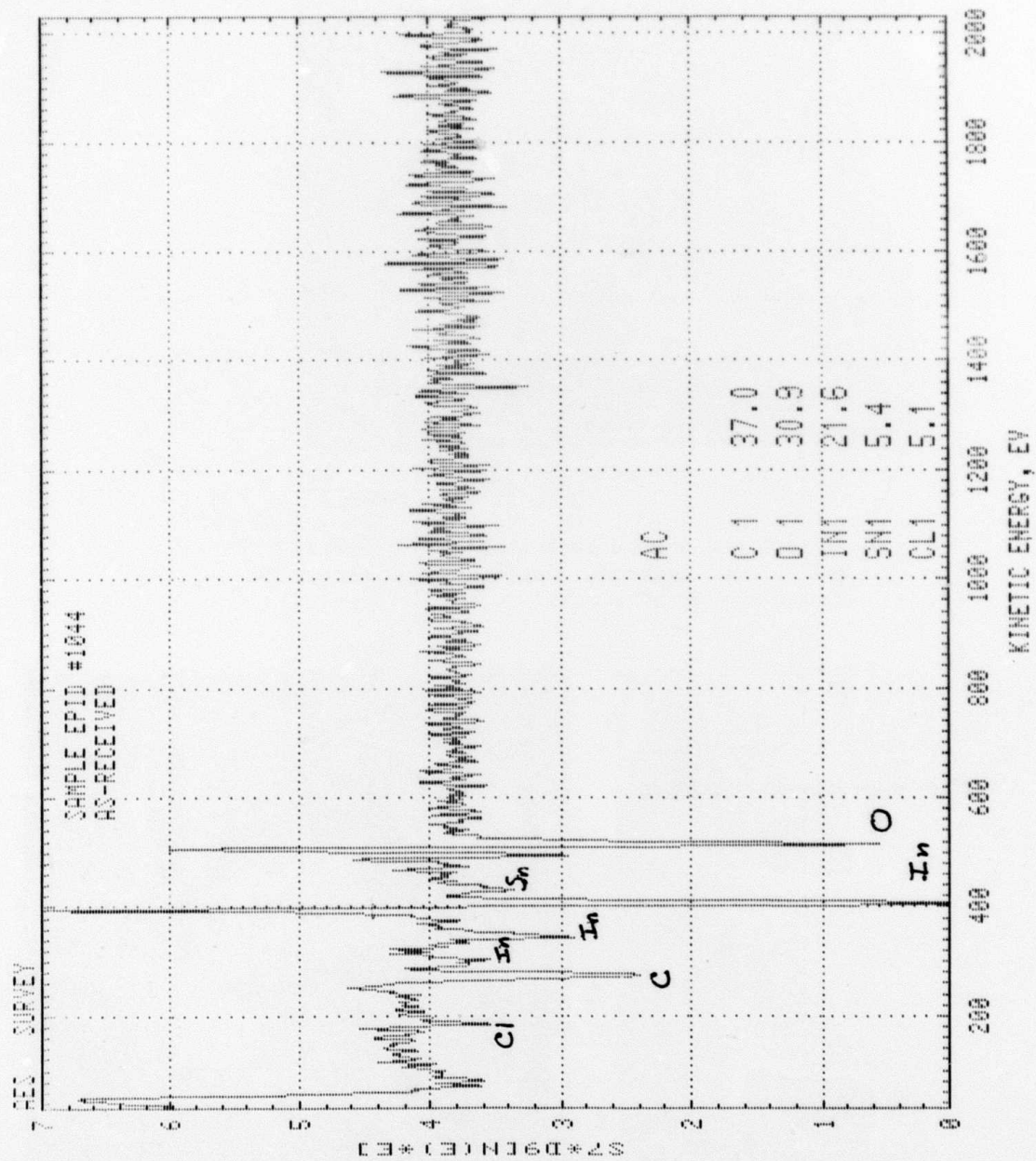


Figure 2: Auger electron spectra for a point on the wall shown in Fig. 1.

Since In and Sn (probably combined with oxygen) are the only conductive elements found on the walls, it can be concluded that redeposition from the row electrode (tin-doped In_2O_3) is responsible for the lowered resistance between the row and column electrodes. The fact that this redeposit is not continuous is consistent with the dependence of resistance on the test voltage. At higher voltages, especially when the suspension is present, conduction across these discontinuities may be possible.

It is the milling of the row electrode at the bottom of the well which is responsible for the redeposit. Since the milling rate is not uniform across the diameter of the beam, the Mylar is completely removed from the center of the substrate before the corners are cleared. Thus, there is considerable over-milling in the centers while milling is completed in the corners. It is expected and observed that the resistance should increase from the center to the edge.

Several steps are being taken to eliminate this low-resistance problem. Additional ion-beam milling at high angles of incidence so that only the walls are milled has been evaluated. Wet etching is also being evaluated. High-angle milling was difficult to control and only partially successful. Etching in dilute aqua regia or dilute buffered hydrofluoric acid has produced better results. However, the aqua regia did attack the evaporated In_2O_3 slightly, especially if the etching was done before removal of the aluminum milling mask. Figure 3 shows the results of etching in dilute buffered Hf; this photo was taken after etching but before the normal ultrasonic rinse. The etch causes the conductive redeposit to be stripped from the walls. Ribbons of redeposit can clearly be seen; these are normally completely removed in a subsequent ultrasonic rinse. Resistance measurements before and after etching indicate removal of the redeposit. It has been noted that devices which have been etched also show less pigment sticking.



Figure 3: Optical micrograph showing ribbons of redeposit removed by etching in dilute buffered hydrofluoric acid.

Redeposit

Means of preventing the formation of the conductive redeposit are also being investigated. Attempts are being made to improve the uniformity of the milling rate. Adjustment of various system parameters has resulted in improvement in the uniformity of the beam current density. It remains to be determined if a sufficiently uniform beam can be obtained so that little or no over-milling would be necessary. The use of a nonconductive layer over the row electrode is being evaluated. SiO_x has been deposited over the row electrodes in attempt to prevent milling of the In_2O_3 . Any overmilling should result in a nonconductive redeposit. Since the added SiO_x on the electrode could prevent proper operation if it were to hold an electrical charge, it will be removed chemically. The dilute buffered Hf has been shown to be satisfactory for this purpose. Devices are being fabricated using this method.

3. PHASE II

3.1 Fabrication Technology

Wet chemical processing is currently being used in the Phase II effort. A process is being developed for the photofabrication of a thick photopolymer layer to serve as the dielectric support for the control grid (columns).

A number of materials have been considered for this dielectric layer. Among them are photoreactive polyimides, photoprintable glass compositions, and various liquid or dry, positive or negative photoresists. While other materials with superior mechanical and chemical properties are still being sought, Shipley 1300 series photoresist has been selected for the initial work.

Since electrical considerations require a relatively thick (12 μm) dielectric layer, a study of coating techniques was required. At first, manual means were used to dispense the photoresist onto the ITO glass. Reasonably satisfactory 12 μm layers were then produced on Phase I size substrates using a Fisher clinical rotator. Suitable coatings on the larger Phase II devices were not achievable with this equipment. Commercial coating machines are generally limited to coating thicknesses of up to about 6-8 μm . Three manufacturers, however, were located who were willing to attempt to provide us with sample coatings of the proper thickness. The first, a spray-coat company, provided us with samples which were either badly "orange peeled" or too thin for our use. A second company provided roller-coated samples. These were only about 7 μm thick, and possessed undesirable features such as excess edge rim and streaks. Finally, smooth 12 μm coatings were produced on a "Cavex" coating machine manufactured by Integrated Technologies, Inc. The machine uses a permeable steel applicator to coat the inverted substrate. Samples off this machine are now being evaluated and processed into EPID cells.

In order to form an intimately contacted photolithographic mask and also to provide the column electrodes, it is necessary to coat the 12 μm dielectric with a conductive layer. Two materials are presently being used to provide this layer. Electron-beam-evaporated aluminum produces a homogeneous, reasonably adherent coating without raising the substrate temperature to the point where the photosensitivity of the resist is destroyed. The major objection to aluminum is its high reflectivity which gives rise to very disturbing spectral reflections in the finished cell. Among the attempts to alleviate this problem was an evaporated In_2O_3 coating. By evaporation from an $\text{In}_2\text{O}_3/\text{In}$ source, a highly conductive film which is opaque to UV yet transmissive to visible light can be deposited on the substrate at low substrate temperatures. This coating reduces the reflectivity substantially.

Both the aluminum and the indium oxide sometimes develop "bubbles" at the interface with the resist which may in turn produce shorts if the bubble breaks away within the cell. Since this bubbling seems to take place when the cell is exposed to light, it is believed that the cause is nitrogen which is liberated from the photoresist during the reaction of light with the photoactive compounds. Tests are underway to determine how to deal with this problem.

The process of etching the conductive layer is essentially in hand. With the exception of occasional wetting problems with the aluminum etch, satisfactory results are being obtained routinely.

Development is done by immersion in Shipley AZ developer. The method of development has been worked out but a few problems remain. The first problem is lifting off of the electrode during development. Much of this problem has to do with over-development of areas on the substrate where the photoresist layer is too thin. This indicates that once the coating technique is perfected, liftoff may be eliminated. Recently, we

have observed a residue which remains after development. This effect appeared concurrently with the cold, dry season, and tests are in progress to determine if relative humidity is the cause.

The cells are being assembled with Mylar spacers and a Devcon epoxy seal. This method works satisfactory but is messy and time consuming. Two types of adhesive gasket materials have been received from Ablestik, Inc. A life test is underway to determine the compatability of the adhesive with the various materials within our cell. The gaskets are available in die-cut shapes and would provide a quick, neat sealing method.

3.2 Results

Recently, eight EPID cells were completed using photoresist as the dielectric. Three cells looked promising while others were useful in the development of the various fabrication processes. The three cells had relatively few defects, produced highly legible characters, and displayed little or no pigment sticking. They did, however, develop "voids" over a period of time. We have not determined whether these voids are due to: leaks, coalition of small surface bubbles left by the capillary filling process or nitrogen which has evolved from the photoresist during exposure to room light.

4. PLANS FOR NEXT QUARTER

- a. Demonstrate a Phase I device to DARPA.
- b. Continue evaluation of Phase II technology.
- c. Obtain masks for Phase II size devices.

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